IS 633 (1985): Specification for DDT Emulsifiable Concentrate [FAD 1: Pesticides and Pesticides Residue Analysis]

BLANK PAGE

IS : 633 - 1985
(Reaffirmed 2012)

Indian Standard

SPECIFICATION FOR DDT EMULSIFIABLE CONCENTRATES

(Second Revision)

UDC 632·951 DDT

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3 July 1985

Indian Standard

SPECIFICATION FOR DDT EMULSIFIABLE CONCENTRATES

(Second Revision)

Pest Control Sectional Committee, AFCDC 6

Chairman	*Representing*
DR K. D. PAHARIA	Directorate of Plant Protection, Quarantine & Storage (Ministry of Agriculture & Rural Development), Faridabad
Members	
DR P. K. AGGARWAL	IDMA Laboratories, Chandigarh
DR S. R. IYENGAR (*Alternate*)	
DR H. L. BAMI	Pesticides Residue Analysis Sectional Committee, AFDC 56, ISI
DR B. BANERJEE	Tea Research Association, Jorhat
DR G. SATYANARAYANA (*Alternate* I)	
SHRI S. C. DAS (*Alternate* II)	
SHRI N. S. BIRDIE	Shriram Test House, Delhi
SHRI R. K. BANERJEE (*Alternate*)	
CHIEF PLANT PROTECTION OFFICER	Department of Agriculture, Government of Maharashtra, Bombay
CHEMIST INCHARAGE, INSECTICIDES TESTING LABORATORY (*Alternate*)	
DR M. S. DHATT	National Malaria Eradication Programme, Delhi
SHRI V. DORAIRAJ	Plastic Containers Sectional Committee, MCPD 11, ISI
SHRI G. D. GOKHALE	Bombay Chemicals Private Limited, Bombay
SHRI V. V. KETKAR (*Alternate*)	
SHRI B. K. VIJAYA GOPAL	Agromore Limited, Bangalore
SHRI S. K. RAMAN (*Alternate*)	
SHRI N. L. KALRA	National Institute of Communicable Diseases, Delhi
SHRI G. C. JOSHI (*Alternate*)	
DR R. L. KALRA	Punjab Agricultural University, Ludhiana
DR R. P. CHAWLA (*Alternate*)	
DR KALYAN SINGH	C. S. A. University of Agriculture & Technology, Kanpur

(*Continued on page 2*)

(*Continued from page* 1)

Members	*Representing*
DR S. K. KASHYAP	National Institute of Occupational Health (ICMR), Ahmadabad
DR S. K. GUPTA (*Alternate*)	
SHRI J. K. KHOSLA	Metal Containers Sectional Committee, MCPD 12, ISI
SHRI A. N. BHATTACHARYYA (*Alternate*)	
SHRI S. G. KRISHNAN	National Organic Chemical Industries Limited, Bombay
DR J. S. VERMA (*Alternate*)	
DR S. LAKSHMANAN	Paper and Flexible Packaging Sectional Committee, MCPD 14, ISI
DR J. C. MAJUMDAR	BASF India Limited, Bombay
DR B. P. CHANDRASEKHAR (*Alternate*)	
SHRI S. K. MAJUMDAR	Central Food Technological Research Institute (CSIR), Mysore
SHRI M. MUTHU (*Alternate*)	
SHRI B. P. S. MANN	Pesticides Association of India, New Delhi
DR B. S. SHETTY (*Alternate* I)	
SHRI T. V. JAGADISAN (*Alternate* II)	
SHRI K. S. MEHTA	Bharat Pulverising Mills Private Limited, Bombay
SHRI S. CHATTERJEE (*Alternate*)	
SHRI J. M. MODI	Pesticides Formulators Association of India (S. S. I.), Bombay
DR S. R. BAROOAH (*Alternate*)	
DR S. K. MUKERJEE	Agricultural Chemicals Division, Indian Agricultural Research Institute (ICAR), New Delhi
DR N. K. ROY (*Alternate*)	
SHRI K. RAJENDRAN NAIR	Central Insecticides Cell, Directorate of Plant Protection, Quarantine and Storage (Ministry of Agriculture & Rural Development), Faridabad
DR R. C. GUPTA (*Alternate* I)	
SHRI S. K. GHOSH (*Alternate* II)	
SHRI P. V. NARAYANAN	Indian Institute of Packaging, Bombay
SHRI K. B. GUPTA (*Alternate*)	
DR V. N. NIGAM	Ministry of Defence (DGI)
SHRI M. M. PADALIA	Department of Agriculture, Government of Gujarat, Ahmadabad
SHRI R. G. JADEJA (*Alternate*)	
SHRI A. R. PANICKER	Hindustan Insecticides Limited, New Delhi
DR S. N. DESHMUKH (*Alternate*)	
SHRI Y. A. PRADHAN	Rallis India Limited, Bombay
SHRI M. L. SHAH (*Alternate*)	
DR R. L. RAJAK	Central Insecticides Laboratory, Directorate of Plant Protection, Quarantine & Storage (Ministry of Agriculture & Rural Development), Faridabad
SHRI V. C. BHARGAVA (*Alternate* I)	
DEPUTY DIRECTOR (PR&QC) (*Alternate* II)	

(*Continued on page 10*)

(*Continued from page* 2)

Members	*Representing*
REPRESENTATIVE	Department of Agriculture, Government of Andhra Pradesh, Hyderabad
DR K. D. SHARMA	Directorate General of Technical Development, New Delhi
SHRI N. R. NARAYANAN (*Alternate*)	
DR K. N. SHRIVASTAVA	Shaw Wallace & Co Limited, Calcutta
SHRI R. N. ROYCHOWDHURY (*Alternate*)	
SHRI A. C. SHROFF	Excel Industries Limited, Bombay
SHRI P. V. KANGO (*Alternate*)	
DR K. SIVASANKARAN	Union Carbide (India) Limited, New Delhi
DR U. V. SINGH (*Alternate*)	
SHRI N. S. VENKATARAMAN	Department of Agriculture, Government of Tamil Nadu, Madras
DR R. V. VENKATARATNAM	Regional Research Laboratory (CSIR), Hyderabad
DR NAGABHUSHAN RAO (*Alternate*)	
DR SANTOSH YELLORE	Analytical Testing Services Pvt Ltd, New Delhi
DR D. B. ANANTHA NARAYANA (*Alternate*)	
SHRI T. PURNANANDAM, Director (Agri & Food)	Director General, ISI (*Ex-officio Member*)

Secretary

SHRI LAJINDER SINGH
Deputy Director (Agri & Food), ISI

AMENDMENT NO, 1 SEPTEMBER 1988
TO
IS : 633-1985 SPECIFICATION FOR DDT EMULSIFIABLE CONCENTRATES

(Second Revision)

(*Page 4, clause 2.2.6*) — Add the following note at the end of the clause:

'NOTE — The material need not be subjected to heat treatment if it has crossed half of its shelf life as ascertained from the data of manufacture and date of expiry declared on the containiner.'

(AFCDC 6)

Printed at Swatantra Bharat Press, Delhi, India

AMENDMENT NO. 2 MAY 1994
TO
IS 633 : 1985 SPECIFICATION FOR DDT EC

(Second Revision)

(Page 4, clause 2.2.5) — Delete.

(Page 6, clause 4.1) — Substitute the following for the existing:

'When freshly manufactured material in bulk quantity is offered for inspection, representative samples of the material shall be drawn and tested as prescribed in IS 10627 : 1983 within 90 days of its manufacture. When the material is offered for inspection after 90 days of its manufacture, sampling shall be done as prescribed in IS 10627. :1983. However, the criteria for conformity of the material when tested, shall be the limits of tolerances, as applicable over the declared nominal value and given under clause 2.3.1 of the standard.'

Indian Standard

SPECIFICATION FOR DDT EMULSIFIABLE CONCENTRATES

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 31 January 1985, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 DDT (Dichloro Diphenyl Trichloroethane) emulsifiable concentrates are largely used in the control of insect pests of medical, veterinary, animal husbandry and agricultural importance.

0.3 This standard was first issued in 1956 and revised in 1975. Experience gained in the manufacture of DDT emulsifiable concentrate formulations in the country pointed towards the need for revision of the existing standard. In this revision the requirements for the various characteristics have been reviewed and brought up-to-date. All the three amendments to 1975 version have also been incorporated.

0.4 DDT emulsifiable concentrates are generally manufactured to contain 25 percent (*m/m*) of DDT, technical.

0.5 In the preparation of this standard due consideration has been given to the provisions of the *Insecticides Act, 1968* and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for DDT emulsifiable concentrates.

*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Constituents — The material shall consist of DDT, technical, dissolved in suitable solvent(s), together with emulsifying agent(s) with or without stabilizer(s) and coupler(s).

2.1.1 DDT, technical, used in the manufacture of DDT emulsifiable concentrates shall conform to IS : 563-1973*.

2.1.2 *Identity* — The material shall comply with identity test as prescribed in Appendix A of IS : 564-1984† and shall not contain any chlorinated pesticide other than DDT technical.

2.2 Physical — The material shall comply with the physical requirements specified in **2.2.1** to **2.2.4.**

2.2.1 *Description* — The material shall be homogeneous and stable liquid, free from sediment. Suspended matter shall be negligible. On dilution with water it shall readily form an emulsion, suitable for spray.

2.2.2 *Cold Test* — No turbidity or separation of solid or oily matter or both shall occur when the material is subjected to the cold test at 10°C as prescribed in **13.1** of IS : 6940-1982‡ or at any other lower temperature as agreed to between the purchaser and the supplier. Introduction of a seeding crystal is not necessary for the test.

2.2.3 *Flash Point (Abel)* — When determined by the method prescribed in IS : 1448 [P : 20]-1984§, the flash point of the material shall be above 24·5°C.

2.2.4 *Emulsion Stability* — Any separation, including creaming at the top and sedimentation at the bottom, of 100 ml of emulsion prepared in standard hard water with 5 ml of concentrate for public health use and with 2 ml of concentrate for agricultural use, shall not exceed 2·0 ml when tested by one of the methods prescribed in **13.3** of IS : 6940-1982‡.

2.2.5 *Heat Stability* — After treating in accordance with the method prescribed in **13.4** of IS : 6940-1982‡ the material shall comply with the requirements specified in **2.2.1** to **2.2.4** and **2.3.1.**

2.3 Chemical — The material shall comply with the chemical requirements specified in **2.3.1** and **2.3.2.**

2.3.1 *DDT, Technical Content* — When determined by the method prescribed in Appendix A, the observed DDT, technical content,

*Specification for DDT, technical (*second revision*).
†Specification for DDT dusting powders (*third revision*).
‡Methods of test for pesticides and their formulations (*first revision*).
§Methods of test for petroleum and its products [P: 20] Flash point by Abel apparatus.

percent by mass, of any of the samples shall not differ from the nominal value by more than the tolerance limits indicated below:

Nominal Value, Percent	*Tolerance Limit, Percent*
Up to 9	+ 10, — 5
Above 9 but below 50	± 5 of the nominal value
50 and above	+ 5, — 3

2.3.1.1 The actual value of DDT, technical content shall be calculated to the second decimal place for rounding off to the first decimal place before applying the tolerance as given in **2.3.1**.

2.3.1.2 The average content of all samples taken shall not be lower than the declared nominal content.

2.3.2 *Acidity or Alkalinity* — When tested by the methods prescribed in **11.3** of IS : 6940-1982*, the acidity, if any, calculated as sulphuric acid (H_2SO_4), or alkalinity, if any, calculated as sodium hydroxide (NaOH), shall not be more than 0·05 percent by mass.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed as per requirements given in IS : 8190 (Part 2)-1980†.

3.2 Marking — The containers shall bear legibly and indelibly the following information in addition to the provisions stipulated under the *Insecticides Act* and Rules:

a) Name of the material;
b) Name of the manufacturer;
c) Date of manufacture;
d) Batch number;
e) Net mass of content;
f) DDT, technical content, percent (*m*/*m*); and
g) The cautionary notice worded as in the *Insecticides Act* and Rules.

3.2.1 Each container may also be marked with the ISI Certification Mark.

---

*Methods of test for pesticides and their formulations (*first revision*).
†Requirements for packing of pesticides : Part 2 Liquid pesticides (*first revision*).

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in IS : 10627-1983*.

5. TESTS

5.1 Tests shall be carried out by the appropriate methods referred to in **2.2.2** to **2.2.4**, **2.3.1** and **2.3.2**.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A
(*Clause* 2.3.1)

DETERMINATION OF DDT, TECHNICAL CONTENT

A-0. GENERAL

A-0.1 There are two methods for the determination of the DDT, technical content, namely, the total organic chlorine method and the hydrolysable chlorine method. Whereas for routine tests both the methods are prescribed, the latter method shall be used as a referee method in case of dispute.

A-1. TOTAL ORGANIC CHLORINE METHOD

A-1.1 Reagents

A.1.1.1 *Isopropyl Alcohol* — of two concentrations, namely, 99 percent, dry, and 50 percent (*v/v*), aqueous solution.

*Methods for sampling of pesticidal formulations.
†Specification for water for general laboratory use (*second revision*).

A-1.1.2 *Metallic Sodium* — pure, in the form of ribbon or cut to small pieces.

A-1.1.3 *Phenolphthalein Indicator Solution* — One percent (*m/v*) in rectified spirit.

A-1.1.4 *Dilute Nitric Acid* — 50 percent by volume.

A-1.1.5 *Standard Silver Nitrate Solution* — 0·1 N.

A-1.1.6 *Ferric Ammonium Sulphate Solution* — Saturated, aqueous, freshly prepared.

A-1.1.7 *Standard Potassium Thiocyanate Solution* — 0·1 N.

A-1.1.8 *Nitric Acid* — Concentrated.

A-1.2 Procedure

A-1.2.1 Weigh accurately a quantity of the material containing about 0·1 g of DDT into a 250-ml Erlenmeyer flask. Add to it 25 ml of isopropyl alcohol (99 percent) and shake the flask to mix the contents. Add to the flask 2·5 g of metallic sodium, connect the flask to a reflux condenser and boil the contents gently for at least 2 hours. Shake the flask occasionally. Dissolve the excess metallic sodium by cautiously adding 10 ml of isopropyl alcohol (50 percent) through the condenser at the rate of 1 to 2 drops per second. Boil for another 10 minutes and then add 60 ml of distilled water.

A-1.2.2 Cool, add 2 to 3 drops of phenolphthalein indicator solution. Neutralize by adding dilute nitric acid dropwise and then add 10 ml in excess. If necessary, cool the flask to room temperature; add a known volume of standard silver nitrate solution in slight excess and coagulate the precipitated silver chloride by digesting on a steam bath for half-an-hour, with frequent stirring. Cool the flask and, if necessary, filter the contents of the flask through a fast qualitative filter paper collecting the filtrate quantitatively in Erlenmeyer flask. Add 5 ml of ferric ammonium sulphate solution either to the cooled unfiltered mixture or to the filtrate, as the case may be, and titrate the excess of silver nitrate with standard potassium thiocyanate solution. (The end point is the appearance of red ferric thiocyanate colour).

A-1.2.3 *Determination of Inorganic Chloride Content* — Weigh accurately about 1 g of the material and transfer with 100 ml distilled water to a 250-ml Erlenmeyer flask. Add 5 ml of concentrated nitric acid and 5 ml of standard silver nitrate solution. Coagulate the precipitate as before and titrate the excess silver nitrate with standard potassium thiocyanate solution. Carry out a blank determination on the reagents as before.

$$\text{Inorganic chlorine content } (c), \text{ percent by mass} = \frac{3{\cdot}546\,(B-A)N}{M}$$

where

B = volume, in ml, of standard potassium thiocyanate solution required for the blank;

A = volume, in ml, of standard potassium thiocyanate solution required for the sample;

N = normality of standard potassium thiocyanate solution; and

M = mass, in g, of the sample taken.

A-1.3 Calculation

$$\text{DDT, technical content, percent by mass} = \frac{7{\cdot}092\ (AN_1 - BN_2)}{M} - 2c$$

where

A = volume, in ml, of standard silver nitrate solution initially taken (*see* **A-1.2.2**);

N_1 = normality of standard silver nitrate solution;

B = volume, in ml, of standard potassium thiocyanate solution used for titration (*see* **A-1.2.2**);

N_2 = normality of standard potassium thiocyanate solution;

M = mass, in g, of the material taken for the test (*see* **A-1.2.1**); and

c = inorganic chlorine content of the material, percent by the mass (*see* **A-1.2.3**).

A-2. HYDROLYSABLE CHLORINE METHOD

A-2.1 Reagents

A-2.1.1 *Acetone*

A-2.1.2 *Alcoholic Potassium Hydroxide Solution* — 1 N.

A-2.1.3 *Nitric Acid* — Approximately 2 N.

A-2.1.4 *Standard Silver Nitrate Solution* — 0·1 N.

A-2.1.5 *Ferric Ammonium Sulphate Solution* — Saturated, aqueous, freshly prepared.

A-2.1.6 *Standard Potassium Thiocyanate Solution* — 0·1 N.

A-2.2 Procedure

A-2.2.1 Weigh accurately a quantity of the material containing about 0·5 g of DDT, into a 250-ml Erlenmeyer flask. Add 50 ml of acetone and 20 ml of alcoholic potassium hydroxide solution, keep it at 20 to 25°C for

15 minutes and then add 50 ml of water. Add to the contents of the flask 20 ml of dilute nitric acid and exactly 25 ml of standard silver nitrate solution. Coagulate the precipitated silver chloride by digesting on a steambath for half-an-hour, with frequent stirring. Cool the flask and, if necessary, filter the contents of flask through a fast qualitative filter paper, collecting the filtrate quantitatively in an Erlenmeyer flask. Add 5 ml of ferric ammonium sulphate solution either to the cooled unfiltered mixture or to the filtrate, as the case may be, and titrate the excess of silver nitrate, with standard potassium thiocyanate solution.

A-2.2.2 Carry out a blank determination using the method given under **A-2.2.1.**

A-2.3 Calculation

$$\text{DDT, technical content, percent by mass} = \frac{35{\cdot}46\ (B - A)\ N}{M} - 10\ c$$

where

B = volume, in ml, of standard potassium thiocyanate solution required for the blank determination (*see* **A-2.2.2**);

A = volume, in ml, of standard potassium thiocyanate solution required for the tests with the material (*see* **A-2.2.1**);

N = normality of standard potassium thiocyanate solution;

M = mass, in g, of the material taken for the test (*see* **A-2.2.1**); and

c = inorganic chlorine content of the material, percent by mass (*see* **A-1.2.3**).

INDIAN STANDARDS INSTITUTION

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones : 3310131, 3311375 Telegrams : Manaksanstha (Common to all offices)

Regional Offices :	*Telephone*
*Western : Manakalaya, E9 MIDC, Marol, Andheri (East), BOMBAY 400093	6 32 92 95
†Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola, CALCUTTA 700054	36 24 99
Southern : C. I. T. Campus, MADRAS 600113	41 24 42
Northern : B69 Phase VII, Industrial Focal Point, S. A. S. NAGAR 160051 (Punjab)	8 73 28
Branch Offices:	
Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMADABAD 380001	2 63 48 2 63 49
'F' Block, Unity Bldg, Narasimharaja Square, BANGALORE 560002	22 48 05
Gangotri Complex, Bhadbhada Road, T. T. Nagar, BHOPAL 462003	6 27 16
22E Kalpana Area, BHUBANESHWAR 751014	5 36 27
5-8-56C L. N. Gupta Marg, HYDERABAD 500001	22 10 83
R14 Yudhister Marg, C Scheme, JAIPUR 302005	6 98 32
117/418 B Sarvodaya Nagar, KANPUR 208005	4 72 92
Patliputra Industrial Estate, PATNA 800013	6 23 05
Hantex Bldg (2nd Floor), Rly Station Road, TRIVANDRUM 695001	32 27
Inspection Office (With Sale Point) :	
Institution of Engineers (India) Building, 1332 Shivaji Nagar, PUNE 411005	5 24 35

*Sales Office in Bombay is at Novelty Chambers, Grant Road, 89 65 28
Bombay 400007

†Sales Office in Calcutta is at 5 Chowringhee Approach, P. O. Princep 27 68 00
Street, Calcutta 700072

Printed at New India Printing Press, Khurja, India